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Indian Standard

METHOD FOR THE DETERMINATION OF MERCURY BY ATOMIC ABSORPTION SPECTROPHOTOMETER

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

**METHOD FOR
THE DETERMINATION OF MERCURY BY
ATOMIC ABSORPTION SPECTROPHOTOMETER**

Chemical Standards Sectional Committee, CDC 1*Chairman**Representing*

DR B. N. MATTOO

Maharashtra State Forensic Science Laboratory,
Bombay*Members*DR S. K. MEGHAL (*Alternate to*
Dr B. N. Mattoo)IDMA Laboratories, Chandigarh
National Organic Chemical Industries Ltd,
Bombay

SHRI P. K. AGARWAL

SHRI K. D. AMRE

SHRI S. B. NANAL (*Alternate*)SHRI J. A. ASHTAPUTRE (*Alternate*)SHRI P. K. RAO (*Alternate*)ASSISTANT DIRECTOR GENERAL (*PFA*)

Ministry of Defence (DGI)

Directorate General of Health Services, New
DelhiASSISTANT SECRETARY (PFA) (*Alternate*)SHRI K. M. BANERJEE (*Alternate*)SHRI N. C. CHATTERJEE (*Alternate*)CHIEF CHEMIST (*Alternate*)Central Revenues Control Laboratory, New
DelhiDEPUTY CHIEF CHEMIST (*Alternate*)JOINT DIRECTOR (CHEMICALS) (*Alternate*)Department of Industries & Commerce,
Government of Tamil Nadu, MadrasDEPUTY DIRECTOR (MCL) (*Alternate*)DR S. K. KAPOOR (*Alternate*)Regional Testing Centre, New Delhi
Shriram Institute for Industrial Research, DelhiDR J. K. NIGAM (*Alternate*)SHRI N. S. BIRDIE (*Alternate*)Sarabhai M. Chemicals, Vadodara
Regional Research Laboratory (CSIR),
HyderabadDR P. K. PANDYA (*Alternate*)DR M. PARDHASARADHI (*Alternate*)Century Rayon, Kalyan
National Physical Laboratory (CSIR), New
DelhiDR J. MADHUSUDAN RAO (*Alternate*)SHRI OM PRASAD (*Alternate*)National Physical Laboratory (CSIR), New
DelhiDR A. K. SARKAR (*Alternate*)*(Continued on page 2)*

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**METHOD FOR
THE DETERMINATION OF MERCURY BY
ATOMIC ABSORPTION SPECTROPHOTOMETER**

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 27 February 1987, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 The atomic absorption spectrophotometric method is based on the fact that atoms present in ground state will absorb incident light of the same wavelength they emit when excited. When radiation from the given excited element is passed through the flame containing atoms of that element in the ground state, the intensity of the transmitted radiation will decrease in proportion to the amount of the ground state atoms present in the flame. A hollow cathode lamp made of the element to be determined provides the radiation. The metal atoms to be measured are introduced into the beam of radiation by aspirating the sample solution into the flame. A monochromator isolates the characteristic radiation from the hollow cathode lamp and photo-sensitive device measures the transmitted radiation. Various elements including mercury can be precisely analyzed by the technique.

0.3 However, flameless absorption analysis making use of hydride technique or a graphite furnace or cold vapour technique are alternatively available. Mercury is frequently determined by cold vapour technique as a free rapid method, which has also been described in the specification.

0.4 Various products, like water and industrial effluents, food products, acids, etc, in which mercury concentration is very low are determined by cold vapour technique.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the method for the determination of mercury by atomic absorption spectrophotometry.

*Rules for rounding off numerical values (*revised*).

2. QUALITY OF THE REAGENTS

2.1 Unless specified otherwise, pure chemicals and deionized water shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. OUTLINE OF THE METHOD

3.1 The sample is brought into solution by suitable treatment with acids or acid combination and diluted suitably with water. Suitable dilutions are made for aspirating into air acetylene flame. The standard solution is prepared in the same way for calibration. The characteristic mercury line is 253.7 mm.

3.2 The cold vapour technique is used for the determination of traces of the mercury. Mercury ions are reduced to the elemental state by suitable reducing agent (see Note). The reduced solution is stirred vigorously so that an equilibrium is achieved between the mercury in solution and air phase. The vapour is then purged into absorption cell which is located in the light path of the spectrometer in place of normal burner. The resultant absorption peak is then recorded.

NOTE — The composition of reducing agent shall be:

- a) Stannous chloride solution — 5 percent (m/v)
- b) Hydrazine hydrate — 40 percent (m/v)
- c) Sodium borohydride — 5 percent (m/v)

4. INTERFERENCE

4.1 Mercury (I) and Mercury (II) show different sensitivities in the air acetylene flame.

Mercury (I) is more sensitive due to absorption reaction $Hg_2^{2+} \rightarrow Hg^{2+} + Hg$. Mercury element is readily atomized with 100 percent efficiency.

4.2 Problem caused by the different sensitivities may be overcome by addition of 1 ml of freshly prepared 20 percent stannous chloride to each solution immediately before analysis. This method is not recommended, unless absolutely necessary, as the elemental mercury formed is readily lost by evaporation. Addition of ascorbic acid has similar effect, while addition of complexing agent (such as EDTA) assists the disproportion reaction by removing divalent mercury.

4.3 In cold vapour technique other metallic ions which are reduced to the elemental state by stannous chloride can interfere. With both flame and vapour technique it is advisable to check for non-atomic absorption by repeating the analysis with hydrogen lamp.

5. APPARATUS

5.1 Atomic Absorption Spectrophotometer — Provided with background correction and having following parameters.

5.1.1 Lamp Current — Depending upon the lamp and the instrument used.

5.1.2 Support — Air.

5.1.3 Fuel — Acetylene.

5.1.4 Flame Stoichiometry — Oxidizing air-acetylene flame.

5.1.5 Wave Length — 253.7 nm.

5.1.6 Band Pass — 0.2 nm.

5.2 Working Range

5.2.1 Air-Acetylene Flame — 100-400 µg/ml.

5.2.2 Cold Vapour Generation Technique — 0.12-1.0 µg/ml.

5.3 Absorption Vessel

5.4 Reaction Vessel

5.5 Magnetic Stirrer

6. REAGENTS

6.1 Pure Mercury — 99.99 percent.

6.2 Concentrated Hydrochloric Acid — See IS : 265-1967*.

6.3 Concentrated Nitric Acid — See IS : 264-1976†.

6.4 Stannous Chloride — Pure.

7. SAMPLE PREPARATION

7.1 A few procedures are given for certain material in which mercury has to be determined for guidance.

7.2 Water and Effluents — Mercury may be present in water as free (ionic) mercury or as organically bound mercury. The latter form is not reduced by stannous chloride during cold vapour technique and more vigorous reduction must be used if total mercury is to be measured.

*Specification for hydrochloric acid (second revision).

†Specification for nitric acid (second revision).

For total mercury estimation, 100 ml sample is digested with 10 ml sulphuric-acid and 5 ml of 2 percent potassium permanganate at minimum temperature for 24 hours. More potassium permanganate may be added in case of sewage or effluent. Alternatively, 5 ml concentrated sulphuric acid, 2·5 ml nitric acid and 1 ml of 5 percent potassium permanganate solution are added to 100 ml of sample. After 15 minutes, 2 ml of 5 percent potassium persulphate solution is added. Excess permanganate is destroyed by just prior to the determination. After the digestion, apply the cold vapour technique.

7.3 Concentration Technique — When mercury content in water or effluent is very low, the concentration of sample is required prior to estimation.

7.3.1 Evaporation — Due to high volatility of mercury compounds, use of evaporation should be avoided.

7.3.2 Solvent Extraction — Mercury may be extracted in methyl isobutyl ketone using 1 percent ammonium pyrrolidine dithiocarbamate in water at pH 3.

7.4 Mercury in Caustic Soda — Select the sample size according to the anticipated mercury concentration and neutralize with (1 : 1) hydrochloric acid using phenolphthalein indicator. Add 1 ml of (1 : 4) sulphuric acid and 1 ml of potassium permanganate. Add 30 ml of water and boil, allow to cool. Destroy the excess potassium permanganate with hydroxylamine hydrochloride solution till colourless. Make up to 100 ml. Take aliquot to the reaction vessel and proceed.

7.5 Mercury in Coal and Coke — Powder the sample to pass through 250 IS sieve and weigh 1 g in pre-ignited crucible. Transfer 10 ml (1 : 9) nitric acid to the combustion bomb, attach the fuse wire to the bomb electrode. Assemble the bomb and charge it with oxygen to 20 atmospheric pressure. Place the bomb in the water bath and ignite the sample. Let it remain for 10 minutes in cold water. Release the pressure and thoroughly rinse the bomb electrodes with 1 percent nitric acid and make to 100 ml. Add potassium permanganate dropwise until permanent colour persists for 60 seconds. Proceed further, as for water and effluent.

7.6 Mercury in Paints — The liquid sample or dried film is transferred into PTEE lined vessel and digested with sulphuric/nitric acid. The digested sample is diluted to known volume with water and mercury is determined by cold vapour technique.

7.7 Mineral Acid and other Inorganic Chemicals — Suitable amount of the sample is diluted to 100 ml and neutralized by adding sodium hydroxide while keeping the beaker in ice. Solution is further diluted to known volume and subsequently suitable aliquot is taken for analysis.

7.8 Inorganic Mercury in Biological Material — Same as stated in 7.2.

8. PROCEDURE

8.1 Air-Acetylene Flame Method — Optimize the response of instrument by adjustment of burner height and flame. Aspirate water to get zero absorbance when stable response is observed, aspirate at least 4 standards and note down absorbance.

Aspirate sample to get absorbance of the sample. Prepare calibration curve by plotting the net absorbance value of the standard against concentration in $\mu\text{g}/\text{ml}$ of mercury. Locate the point of the sample absorbance and calculate the concentration of mercury in the sample. Background correction may be made in all the readings with hydrogen continuum lamp. The net reading should be taken for calculation.

8.2 Vapour Generation Technique — Place the absorption cell of the apparatus on the spectrophotometer burner mount. Connect the reaction vessel to the cell with appropriate tubing. Place 50 ml aliquot of the sample in the reaction vessel (if mercury content is higher, take less sample and add water to level 50 ml in reaction vessel). Add 1 ml of 20 percent stannous chloride solution and immediately stopper the vessel. Stir the solution vigorously for ninety seconds. Turn off the stirrer, turn on the air valve (air flow 4 litre/min) and purge the vapour into the absorption cell. A sharp analytical peak will be recorded immediately. This could better be carried out on peak-hold mode.

Take at least 3 standard readings and take replicate reading of the samples. Correct non-atomic absorption, if necessary, and calculate the mercury in sample. Take a reagent blank reading.

9. CALCULATION

$$\text{Mercury, percentage by mass} = \frac{C \times V}{10^6} \times \frac{100}{M}$$

where

C = concentration of mercury in $\mu\text{g}/\text{ml}$ in final solution,

V = volume in ml of final solution, and

M = mass in g of sample in final solution.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>	<i>Definition</i>
Force	newton	N	$1 \text{ N} = 1 \text{ kg}\cdot\text{m/s}^2$
Energy	joule	J	$1 \text{ J} = 1 \text{ N}\cdot\text{m}$
Power	watt	W	$1 \text{ W} = 1 \text{ J/s}$
Flux	weber	Wb	$1 \text{ Wb} = 1 \text{ V}\cdot\text{s}$
Flux density	tesla	T	$1 \text{ T} = 1 \text{ Wb/m}^2$
Frequency	hertz	Hz	$1 \text{ Hz} = 1 \text{ c/s(s}^{-1}\text{)}$
Electric conductance	siemens	S	$1 \text{ S} = 1 \text{ A/V}$
Electromotive force	volt	V	$1 \text{ V} = 1 \text{ W/A}$
Pressure, stress	pascal	Pa	$1 \text{ Pa} = 1 \text{ N/m}^2$